

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
11 October 2001 (11.10.2001)

PCT

(10) International Publication Number
WO 01/74306 A2

(51) International Patent Classification⁷: **A61K 7/00**

TANG, Xiaozhong; 190 Crestview Road, Bridgewater, NJ 08807 (US).

(21) International Application Number: PCT/US01/10330

(74) Agent: **MIANO, Rosemary, M.**; Colgate-Palmolive Company, 909 River Road, P.O. Box 1343, Piscataway, NJ 08855-1343 (US).

(22) International Filing Date: 29 March 2001 (29.03.2001)

(25) Filing Language: English

(81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.

(26) Publication Language: English

(30) Priority Data:
60/194,373 4 April 2000 (04.04.2000) US
09/712,378 14 November 2000 (14.11.2000) US

(71) Applicant: **COLGATE-PALMOLIVE COMPANY**
[US/US]; 300 Park Avenue, New York, NY 10022 (US).

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

(72) Inventors: **MATTAI, Jairajh**; 280 River Road, Apt. 21B, Piscataway, NJ 08854 (US). **GUENIN, Eric, P.**; 14 Arvida Drive, Pennington, NJ 08534 (US). **AFFLITTO, John**; 2 Jay Drive, Brookside, NJ 07926 (US). **HOGAN, John**; 242B Pleasantview Drive, Piscataway, NJ 08854 (US). **JONAS, John**; 96 West End Avenue, Summit, NJ 07901-1222 (US). **LEE, Wilson**; 51 Hearthstone Road, Bloomfield, NJ 07003 (US). **LINN, Elizabeth**; 621 Fifth Avenue, Lyndhurst, NJ 07071 (US). **MUNSAYAC, Rosemary**; 5 Tenney Road, West Orange, NJ 07052 (US).

Published:

— *without international search report and to be republished upon receipt of that report*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: STABLE AND EFFICACIOUS SOFT SOLID PRODUCT

(57) Abstract: A low residue, stable antiperspirant and/or deodorant composition is disclosed which comprises: (a) a dimethicone/vinyldimethicone crosspolymer composition made by reacting a polymethylhydrogensiloxane with an alpha, omega-divinylpolydimethyl siloxane for which the dimethicone/vinyldimethicone crosspolymer composition is used at a concentration of 0.5-10 % in cyclomethicone; (b) polyethylene beads having a density in the range of 0.91-0.98 grams/cm³ and a particle size in the range of 5-40 microns, wherein the polyethylene beads are used in an amount of at least 2 % by weight based on the total weight of the composition; (c) a volatile silicone; (d) an emollient or mixture of two or more emollients; and (e) an effective amount of an antiperspirant active material.

WO 01/74306 A2

STABLE AND EFFICACIOUS SOFT SOLID PRODUCT

Field of the Invention

5 This invention relates to soft solid products made as suspensions and suitable for use as antiperspirants and/or deodorants.

Background of the Invention

There is a continuing trend to develop new and superior cosmetic compositions especially for the reduction and/or elimination of wetness and/or odor under the arms. 10 Particular efforts include developing lower residue products especially with improved efficacy and aesthetics. Various product forms have included sticks (especially gel/sticks), gels, soft solids, roll-ons, aerosols and creams. Of these various forms the sticks, gels, soft solids creams and roll-ons are made with a liquid base material incorporating a solidifying agent and/or gelling agent and/or thickening agent. 15 Generally, these forms include a solution of the cosmetically active ingredient in a suitable vehicle, a suspension of the active ingredient in a carrier vehicle, or a multiphasic dispersion or emulsion in which a solution of the active ingredient is dispersed or suspended in some continuous phase or in which the solubilized active ingredient constitutes the continuous phase.

20 A variety of soft-solid formulations are known. These include formulations made with the following ingredients:

(a) clay thickening agent and an activator for the clay: for example, U.S. Patent Number 5,019,375 to Tanner et al; and U.S. Patent Number 4,526,780 to Marschner et al;

25 (b) particulate thickening agents such as fumed silica: for example, U.S. Patent Number 5,069,897 to Orr; and U. S. Patent Number 4,937,069 to Shin;

(c) selected volatile and/or non-volatile alkylmethylsiloxanes such as those including a structuring wax: for example, U.S. Patent Number 5,225,188 to Abrutyn et al; and PCT applications WO 97/16161 and 16162 both of which are assigned to 30 Unilever PLC; and

(d) triglyceride gellants such as the glyceryl tribehenate described in U.S. Patent Number 5,718,890 to Putnam et al.

The use of a class of compositions known as silicone elastomers in cosmetic compositions has shown some interesting results. PCT case WO 97/44010 and 35 assigned to the same assignee as this application describes a silicone gel material made by combining (a) a volatile silicone material and (b) an organopolysiloxane material (or silicone elastomer) as a gelling agent wherein the organopolysiloxane material (silicone

elastomer) can be a reaction product of a vinyl-terminated siloxane polymer and a silicon hydride cross-linking agent. Related technology is also disclosed in PCT case WO 98/00097, WO 98/00104 and 98/00105 assigned to Unilever PLC on cross-linked non-emulsifying elastomers.

5 U.S. Patent 5,599,533 to Stepniewski et al assigned to Estee Lauder describes a stable water-in-oil emulsion system formed with an organopolysiloxane elastomer, a vehicle in which the elastomer is dispersed or dispersible, a stabilizing agent, a surfactant and an aqueous component. A commercial product known as "REVELATION" retexturizing complex for hands and chest sold by the same assignee
10 contains a silicone gel material with an organopolysiloxane component and octamethylcyclotetrasiloxane.

EP 0 787 758 A1 teaches a method for solvent thickening by using a silicone latex having a plurality of crosslinked polysiloxane particles.

Another recent case assigned to the same assignee as this application is WO
15 99/51192 and U.S. Patent Application Serial Number 9/273152 which describes antiperspirant compositions with the use of broad categories of elastomers. Other examples of the use of elastomer type materials and/or methods for processing such materials may be found in PCT cases WO 98/00097; WO 98/00104; WO 98/00105; WO 98/18438; WO 98/42307 all of which are incorporated herein by reference.

20 Two major problems have been observed when the use of elastomer materials is included in soft solid formulations. The first problem is reduction in efficacy due to the formation of an occlusive elastomeric film which prevents the active from diffusing into the sweat duct. The second problem is the consistency of the product as evidenced by high viscosity and elastic behavior when applied to the surface of the skin. In order
25 to reduce this high viscosity, emollients and solvents have to be added which may negatively impact efficacy of the deodorant and/or antiperspirant. It has been found that the use of a selected type of elastomer in soft solid formulations in combination with polyethylene beads as described herein overcomes these problems.

Thus, it is an object of the invention to provide improved cosmetic
30 compositions with the improvements as previously described and which are useful as antiperspirants and/or deodorants. These and other objects of the invention will be apparent from the following description of the invention.

35

Summary of the Invention

It has been found that an improved soft solid cosmetic product may be made as a suspension formed with:

5 (a) a dimethicone/vinyldimethicone crosspolymer composition made by reacting (in the presence of a platinum catalyst) a polymethylhydrogensiloxane with an alpha, omega-divinylpolydimethyl siloxane for which the dimethicone/vinyldimethicone crosspolymer composition (1) is used at a concentration of 4-10% in cyclomethicone (particularly 4-7%, and, more particularly, 4-6.5%) (for example, where the cyclomethicone is a D4 or D5 cyclomethicone), (2) has a refractive index in the range
10 of 1.392-1.402 at 25 degrees C, and (3) has a viscosity in the range of $0.013 - 1 \times 10^4$ Pascal seconds;

(b) polyethylene beads having a density in the range of 0.91-0.98 grams/cm³ and a particle size in the range of 5-40 microns, wherein the polyethylene beads are used in an amount of at least 2% by weight based on the total weight of the composition;

15 (c) a volatile silicone;

(d) an emollient (which may also be a mixture of two or more emollients and which may include a non-volatile silicone); and

(e) an effective amount of an antiperspirant active material to cause an antiperspirant and/or a deodorant effect.

20 The soft solid antiperspirant and/or deodorant product of this invention is an opaque product which leaves little or no white residue when applied and which exhibits improved efficacy and stability as compared to other formulations with different types of elastomers. Reduction of sweat of at least 40% can be achieved with the compositions of the invention as compared to typical levels of 15% for other products.

25 Detailed Description of the Invention

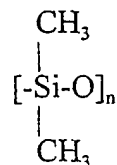
The stable, high efficacy, low residue cosmetic compositions of this invention are made by combining:

- (a) 40-75% (particularly 45-60%, and, more particularly, 46-53%) of a volatile silicone (especially a D5 cyclomethicone);
- 30 (b) 0.1-20% (particularly 2-18% and, more particularly, 5-15%) of an emollient or a mixture of two or more emollients (for example, 0.1-5% (particularly 0.3-4.0%, more particularly 0.4-2.0% and even more particularly 0.4-1.5%) of a non-volatile silicone such as phenyltrimethicone; or 1-12% C12-15 alkyl benzoate);
- (c) 0.5-6% (on a solids basis) (particularly 1-6% and, more particularly, 2-5%) of
35 the dimethicone/vinyldimethicone crosspolymer composition as described above;

- (d) 0.1-20% (particularly 10-20% to get an antiperspirant effect) of an antiperspirant active based on an anhydrous, buffer-free antiperspirant active;
- (e) 2-15% (particularly 2-10% and, more particularly, 2-8%) of polyethylene beads having a particle size in the range of 5-40 microns and a density in the range of 0.91-0.98 g/cm³;
- (f) 0-5% antimicrobial agent; and
- (g) 0-5% fragrance;

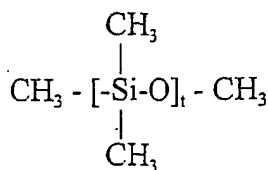
wherein all percents are in percents by weight based on the total weight of the composition unless otherwise indicated.

The silicone materials used in forming the compositions of the present invention may be selected from the group consisting of conventional cyclic and linear volatile and non-volatile silicones which act as a swelling agent for the suitable elastomer. Illustratively, and not by way of limitation, the volatile silicones are one or more members selected from the group consisting of cyclic polydimethylsiloxanes such as those represented by Formula I:



Formula I

where n is an integer with a value of 3-7, particularly 5-6. By volatile silicone material is meant a material that has a measurable vapor pressure at ambient temperature. For example, DC-245 fluid from Dow Corning Corporation (Midland, Michigan) is a type of cyclomethicone which can be used. These include a tetramer (or octylmethylcyclotetrasiloxane) and a pentamer (or decamethylcyclopentasiloxane). The nonvolatile and volatile linear silicones are one or more members selected from the group consisting of linear polydimethylsiloxanes such as those represented by Formula II:



Formula II

and t is selected so that the molecular weight ranges from 800-260,000 and the viscosity ranges from 5-600,000 centistokes, for example Dimethicone DC 200 from Dow Corning.

Emollients are a known class of materials in this art, imparting a soothing effect to the skin. These are ingredients which help to maintain the soft, smooth, and pliable appearance of the skin. Emollients are also known to reduce whitening on the skin and/or improve aesthetics. Examples of chemical classes from which suitable emollients can be found include:

(a) fats and oils which are the glyceryl esters of fatty acids, or triglycerides, normally found in animal and plant tissues, including those which have been hydrogenated to reduce or eliminate unsaturation. Also included are synthetically prepared esters of glycerin and fatty acids. Isolated and purified fatty acids can be esterified with glycerin to yield mono-, di-, and triglycerides. These are relatively pure fats which differ only slightly from the fats and oils found in nature. The general structure may be represented by Formula III:



Formula III

wherein each of R^1 , R^2 , and R^3 may be the same or different and have a carbon chain length (saturated or unsaturated) of 7 to 30. Specific examples include peanut oil, sesame oil, avocado oil, coconut, cocoa butter, almond oil, safflower oil, corn oil, cotton seed oil, castor oil, hydrogenated castor oil, olive oil, jojoba oil, cod liver oil, palm oil, soybean oil, wheat germ oil, linseed oil, and sunflower seed oil;

(b) hydrocarbons which are a group of compounds containing only carbon and hydrogen. These are derived from petrochemicals. Their structures can vary widely and include aliphatic, alicyclic and aromatic compounds. Specific examples include paraffin, petrolatum, hydrogenated polyisobutene, and mineral oil.

(c) esters which chemically, are the covalent compounds formed between acids and alcohols. Esters can be formed from almost all acids (carboxylic and inorganic) and any alcohol. Esters here are derived from carboxylic acids and an alcohol. The general structure would be $R^4\text{CO-OR}^5$. The chain length for R^4 and R^5 can vary from 7 to 30 and can be saturated or unsaturated, straight chained or branched. Specific examples include isopropyl myristate, isopropyl palmitate, isopropyl stearate, isopropyl isostearate, butyl stearate, octyl stearate, hexyl laurate, cetyl stearate, diisopropyl adipate, isodecyl oleate, diisopropyl sebacate, isostearyl lactate, C_{12-15} alkyl

benzoates, myreth-3 myristate, dioctyl malate, neopentyl glycol diheptanoate, neopentyl glycol dioctanoate, dipropylene glycol dibenzoate, C₁₂₋₁₅ alcohols lactate, isohexyl decanoate, isohexyl caprate, diethylene glycol dioctanoate, octyl isononanoate, isodecyl octanoate, diethylene glycol diisononanoate, isononyl isononanoate, isostearyl isostearate, behenyl behenate, C₁₂₋₁₅ alkyl fumarate, laureth-2 benzoate, propylene glycol isoceteth-3 acetate, propylene glycol ceteth-3 acetate, octyldodecyl myristate, cetyl ricinoleate, myristyl myristate.

(d) saturated and unsaturated fatty acids which are the carboxylic acids obtained by hydrolysis of animal or vegetable fats and oils. These have general structure R⁶COOH with the R⁶ group having a carbon chain length between 7 and 30, straight chain or branched. Specific examples include lauric, myristic, palmitic, stearic, oleic, linoleic and behenic acid.

(e) saturated and unsaturated fatty alcohols (including guerbet alcohols) with general structure R⁷COH where R⁷ can be straight or branched and have carbon length of 7 to 30. Specific examples include lauryl, myristyl, cetyl, isocetyl, stearyl, isostearyl, oleyl, ricinoleyl and erucyl alcohol;

(f) lanolin and its derivatives which are a complex esterified mixture of high molecular weight esters of (hydroxylated) fatty acids with aliphatic and alicyclic alcohols and sterols. General structures would include R⁸CH₂-(OCH₂CH₂)_nOH where R⁸ represents the fatty groups derived from lanolin and n=5 to 75 or R⁹CO-(OCH₂CH₂)_nOH where R⁹CO- represents the fatty acids derived from lanolin and n=5 to 100. Specific examples include lanolin, lanolin oil, lanolin wax, lanolin alcohols, lanolin fatty acids, isopropyl lanolate, ethoxylated lanolin and acetylated lanolin alcohols.

(g) alkoxylated alcohols wherein the alcohol portion is selected from aliphatic alcohols having 2-18 and more particularly 4-18 carbons, and the alkylene portion is selected from the group consisting of ethylene oxide, and propylene oxide having a number of alkylene oxide units from 2-53 and, more particularly, from 2-15. Specific examples include PPG-14 butyl ether and PPG-53 butyl ether.

(h) silicones and silanes the linear organo-substituted polysiloxanes which are polymers of silicon/oxygen with general structure:

(1) (R¹⁰)₃SiO(Si(R¹¹)₂O)_xSi(R¹²)₃ where R¹⁰, R¹¹ and R¹² can be the same or different and are each independently selected from the group consisting of phenyl and C1-C60 alkyl;

(2) HO(R¹⁴)₂SiO(Si(R¹⁵)₂O)_xSi(R¹⁶)₂OH, where R¹⁴, R¹⁵ and R¹⁶ can be the same or different and are each independently selected from the group consisting of phenyl and C1-C60 alkyl; or

(3) organo substituted silicon compounds of formula $R^{17}Si(R^{18})OSiR^{19}$ which are not polymeric where R^{17} , R^{18} and R^{19} can be the same or different and are each independently selected from the group consisting of phenyl and C1-C60 alkyl optionally with one or both of the terminal R groups also containing an hydroxyl group. Specific examples include dimethicone, dimethiconol behenate, C_{30-45} alkyl methicone, stearoxytrimethylsilane, phenyl trimethicone and stearyl dimethicone.

(i) mixtures and blends of two or more of the foregoing.

Emollients of special interest include C12-15 alkyl benzoate (FINSOLV TN from Finetex Inc., Elmwood Park, NJ), isopropyl myristate; and neopentyl glycol diheptanoate.

The emollient or emollient mixture or blend thereof incorporated in compositions according to the present invention can, illustratively, be included in amounts of 0.5 - 50 %, preferably 1 - 25 %, more preferably 3 - 12 %, by weight, of the total weight of the composition.

One particular elastomer of interest is KSG-15 silicone elastomer from Shin-Etsu Silicones of America (Akron, Ohio).

The antiperspirant active can be selected from the group consisting of any of the known antiperspirant active materials. These include, by way of example (and not of a limiting nature), aluminum chlorohydrate, aluminum chloride, aluminum sesquichlorohydrate, zirconyl hydroxychloride, aluminum-zirconium glycine complex (for example, aluminum zirconium trichlorohydrate gly, aluminum zirconium pentachlorohydrate gly, aluminum zirconium tetrachlorohydrate gly and aluminum zirconium octochlorohydrate gly), aluminum chlorohydrate PG, aluminum chlorohydrate PEG, aluminum dichlorohydrate PG, and aluminum dichlorohydrate PEG. The aluminum-containing materials can be commonly referred to as antiperspirant active aluminum salts. Generally, the foregoing metal antiperspirant active materials are antiperspirant active metal salts. In the embodiments which are antiperspirant compositions according to the present invention, such compositions need not include aluminum-containing metal salts, and can include other antiperspirant active materials, including other antiperspirant active metal salts. Generally, Category I active antiperspirant ingredients listed in the Food and Drug Administration's Monograph on antiperspirant drugs for over-the-counter human use can be used. In addition, any new drug, not listed in the Monograph, such as aluminum nitrate hydrate and its combination with zirconyl hydroxychlorides and nitrides, or aluminum-stannous chlorohydrates, can be incorporated as an antiperspirant active ingredient in antiperspirant compositions according to the present invention.

Particular types of antiperspirant actives include aluminum zirconium trichlorohydrate and aluminum zirconium tetrachlorohydrate either with or without glycine. A particular antiperspirant active is aluminum trichlorohydrate gly such as AZZ-902 SUF (from Reheis Inc., Berkeley Heights, NJ) which has 98% of the particles less than 10 microns in size.

Antiperspirant actives can be incorporated into compositions according to the present invention in amounts in the range of 0.1 – 25% of the final composition, but the amount used will depend on the formulation of the composition. For example, at amounts in the lower end of the broader range (for example, 0.1 - 10% on an actives basis), a deodorant effect may be observed. At lower levels the antiperspirant active material will not substantially reduce the flow of perspiration, but will reduce malodor, for example, by acting as an antimicrobial material. At amounts of 10-25% (on an actives basis) such as 15 - 25%, by weight, of the total weight of the composition, an antiperspirant effect may be observed.

The antiperspirant active material is desirably included as particulate matter suspended in the composition of the present invention in amounts as described above, but can also be added as solutions or added directly to the mixture.

The polyethylene beads useful with this invention have a density in the range of 0.91-0.98 g/cm³ and a particle size in the range of 5-40 microns, with one particular type of polyethylene having a particle size of 20 microns. All particle sizes are averages. Several types of suitable polyethylene beads that are commercially available are MICROTHENE FN 510 from Equistar Chemicals LP (Houston, Texas); ACUMIST A-6 from Allied Signal Corp., Morristown, NJ), and Performalene™ (New Phase Technology, Piscataway, NJ). It is believed that the polyethylene component contributes to the reduction in syneresis and is also responsible for giving the products a powdery feel as determined by trained sensory panels.

Suitable antimicrobial agents include, for example, bacteriostatic quaternary ammonium compounds such as 2-amino-2-methyl-1-propanol (AMP), cetyltrimethylammonium bromide, cetyl pyridinium chloride, 2, 4, 4'-trichloro-2'-hydroxydiphenylether (Triclosan), N-(4-chlorophenyl)-N'-(3,4-dichlorophenyl)urea (Triclocarban), silver halides, octoxyglycerin (Sensiva™ SC 50) and various zinc salts (for example, zinc ricinoleate). The bacteriostat can, illustratively, be included in the composition in an amount of 0-5%, particularly 0.01-1.0% by weight, of the total weight of the composition. Triclosan, can illustratively be included in an amount of from 0.05% to about 0.5% by weight, of the total weight of the composition.

A variety of fragrances can be used in these compositions if a scented products is desired. Fragrances can be used in an amount in the range of 0-5%, particularly 0.01-2.0%, and, for example, at a level of 1%.

Masking agents can be used in an amount of 0.05-5.0% (particularly 0.05-2%)
5 by weight based on the total weight of the composition if an unscented product is desired.

Other various optional components include those described in U.S. Patents Numbers 5,019,375 to Tanner et al; 4,937,069 to Shin; and 5,102,656, each of which is incorporated by reference in its entirety herein. Examples of such additional
10 ingredients include fragrances, coloring agents, opacifiers, etc. in types and amounts conventionally used for such products.

These compositions are soft solids made as suspensions and thickened or gelled by the elastomer component. While other thickeners may be used, the compositions of this invention will normally use the elastomer component as the only gellant. Of course
15 various viscosities for a soft solid may be made depending on the amount of elastomer material and the amount of other ingredients used. One group having a more viscous form will have a viscosity in the range of 25,000-2,000,000 centipoise, particularly 50,000 - 1,000,000 centipoise, and suitable for use with an applicator with porous openings or slots such as those described in USSN 9/191,897 (PCT 99/25570).
20 incorporated by reference herein as to the description of the applicators. Another form will have a lower viscosity such as in the range of 20,000-200,000 centipoise and will be suitable for use with applicators requiring a thinner composition, for example roll-on applicators which have a rolling ball structure. For example, such roll-on applicators are described in U.S. Patents Numbers 5,158,385 and 4,984,921. incorporated by
25 reference herein as to the description of the applicators.

While various forms have been described, it is believed that the compositions made according to this inventions should preferably have a ratio of elastomer to antiperspirant active in the range of 1:2-1:20 in order to achieve the optimum improved efficacy and the improved stability that has been observed.

30 Compositions according to the present invention can be made by mixing the silicone gel material with surfactant(s), active ingredient(s) and optionally one or more of emollient(s), thickener(s) and fragrance. Mixing conditions and the use of heating will depend on what types of materials are being combined and; the melting points for those materials as are known to those skilled in the art. For example if soft solids, roll-
35 ons or gels are being made, temperatures, in the range of room temperature or slightly higher (for example, 25-50 degrees C, particularly 23-30 degrees C) may be used. For stick products and soft solid/cream products made with higher melting point materials

(for example, high temperature waxes) temperatures from 25-85 degrees C may be used. The mixture can be introduced into dispensing containers known to those skilled in the art including those for solids, gels, roll-ons, soft solids and creams. In one particular example, slotted dispensers may be used such as those known in the art, for example, those having a parallel row or rows of straight or curved slots or holes with a screw mechanism for forcing the composition through the top as the product is used.

Where the dispensing containers have a top surface with slots therein, the composition can be rubbed onto the skin from the top surface of the container (itself fed from a reservoir of product in the container) so as to deposit an adequate amount of the cosmetic composition on to the skin. The cosmetic composition, for example, an antiperspirant and/or deodorant in the form of a soft solid, can be extruded from inside the dispensing container through the slots or holes onto the top of the surface of the dispensing container, and from there may be applied to the skin in the axillary regions to deposit sufficient amounts of antiperspirant and/or deodorant active material to reduce body malodor and/or reduce perspiration in axillary regions of the human body.

Various forms of the invention can be exemplified by the following formulations but should not be construed as limitations on the invention:

Soft Solid

- (a) 1-20% of a volatile silicone such as D4 or D5 cyclomethicone (or mixtures thereof);
- (b) an emollient component comprising 0.1-8% of FINSOLV TN C12-15 alkyl benzoate, 1-8% neopentyl glycol diheptanoate, 0.5-2% isopropyl myristate and 0.1-1.5% of a non-volatile silicone such as phenyltrimethicone;
- (c) 40-60% elastomer composition (KSG-15 elastomer composition);
- (d) 15-25% antiperspirant active;
- (e) 3-10% polyethylene beads of the type described above; and
- (f) optionally 0.5-1.5% fragrance.

EXAMPLES

The following Examples are offered as illustrative of the invention and are not to be construed as limitations thereon. In the Examples and elsewhere in the description of the invention, chemical symbols and terminology have their usual and customary meanings. In the Examples as elsewhere in this application (a) values for n, m, etc. in formulas, molecular weights and degree of ethoxylation or propoxylation are averages; (b) temperatures are in degrees C unless otherwise indicated; and (c) the amounts of the components are in weight percents based on the standard described; if no other standard is described then the total weight of the composition is to be inferred. Various names of chemical components include those listed in the CTFA International

Cosmetic Ingredient Dictionary (Cosmetics, Toiletry and Fragrance Association, Inc., 7th ed. 1997). Mixing techniques used to make the compositions are those conventionally used in the art including those described above.

5

Example 1: General Method of Making Compositions

The solvent components such as the volatile silicone (for example, cyclomethicone), emollients (such as non-volatile silicone (for example, phenyltrimethicone), C12-15 alkyl benzoate, neopentyl glycol diheptanoate and isopropyl myristate) are added to a large capacity mixer equipped with a mechanical stirrer and blended for about 5 minutes or until a homogeneous dispersion is formed. The antiperspirant active is then added as a dry powder with continuous mixing followed by the polyethylene beads. The entire mixture is mixed for about 20 minutes or until a homogeneous dispersion is formed. The sides of the mixing vessel are scraped with a spatula of sufficient size to free solid chunks of particulates. The elastomer is then added and blending is continued for about 20 minutes or until a homogeneous white creamy paste is formed. If fragrance is added it is done so at this point and mixed until well blended or for about 5 minutes. The resulting soft solid is then passed through a homogenizer (KINEMATIC homogenizer) and placed back into the reaction vessel. Multiple passes through the homogenizer are used (usually about 1-4) until the product is stable, that is, exhibits a syneresis of less than 8%, especially less than 5% as evaluated by the process described in Example 3, below. No heating steps are required by this process. If, however, a component is used which requires melting, the premelting of this component will be required.

25

Examples 1A, 3A and 4A

The method of Example 1 was used to make the following compositions with the amounts of ingredients shown in TABLE A. The numbers are weight percent based on the total weight of the composition.

30

35

TABLE A

Ingredient	Ex. 1A	Ex. 3A	Ex. 4A
Elastomer (KSG-15 polymer from Shin-Etsu)	47.50	49.50	47.50
Antiperspirant active (AZZ-902 SUF)	25.00	25.00	25.00
Phenyl trimethicone (Dow Corning 556)	1.00	1.00	0
C12-15 alkyl benzoate (FINSOLV TN)	5.00	3.00	5.00
Neopentyl glycol diheptanoate	5.00	5.00	3.00
Cyclomethicone (Dow Corning 245)	8.50	8.50	10.50
Isopropyl myristate	1.00	1.00	1.00
Polyethylene (MICROTHENE FN 510)	6.00	6.00	7.00
Fragrance	1.00	1.00	1.00
Total	100%	100%	100%

Examples 5A-10A

- 5 The method of Example 1 was used to make the following compositions with the amounts of ingredients shown in TABLES B and C. The numbers are weight percent based on the total weight of the composition.

TABLE B

Ingredient	Ex. 5A	Ex. 6A	Ex. 7A	Ex. 8A
Neopentyl glycol diheptanoate	1.00	3.00	3.00	3.00
Cyclomethicone (DC-245)	10.00	10.50	11.50	11.50
Phenyl trimethicone (DC 556)	1.00	1.00		1.00
Isopropyl myristate	1.00	1.00	1.00	
C12-15 alkyl benzoate (Finsolv TN)	5.00	5.00	5.00	5.00
AlZr trichlorohydrex gly (Reach AZZ 902 SUF)	25.00	25.00	25.00	25.00
Polyethylene (Microthene FN 510)	6.00	6.00	6.00	6.00
Elastomer (KSG-15 from Shin-Etsu)	50.00	47.50	47.50	47.50
Fragrance	1.00	1.00	1.00	1.00
Total	100.00	100.00	100.00	100.00

10

15

20

TABLE C

Ingredients	Ex.9A	Ex.10A
Neopentyl glycol diheptanoate		
Cyclomethicone (DC-245)	12.50	21.50
Phenyl trimethicone (DC 556)	1.00	
Isopropyl myristate	1.00	
C12-15 alkyl benzoate (Finsolv TN)	5.00	
AlZr trichlorohydrex gly (Reach AZZ 902 SUF)	25.00	25.00
Polyethylene (Microthene FN 510)	8.00	6.00
Elastomer (KSG-15 from Shin-Etsu)	47.50	47.50
Fragrance		
Total	100.00	100.00

5

Example 2: Alternative General Method of Making Compositions

The elastomer composition is weighed out in a stainless steel container, which container is part of a Hobart Mixer (Model N-50, from Hobart Corporation, Troy, OH). The cyclomethicone, dimethicone, neopentyl glycol diheptanoate, diethyl phthalate and isopropyl myristate are weighed separately and added sequentially to the same

10 container as the elastomer. The ingredients are blended for about 5 minutes or until a homogeneous mixture is formed. The antiperspirant active is then slowly added while mixing is continued. Mixing is then continued for an additional 10 minutes. Next the polyethylene powder is added slowly with mixing. After the addition is completed, mixing is continued for an additional 10 minutes. If fragrance is used it is added at this

15 point and mixing is continued for an additional 5 minutes. The soft solid product is then transferred to suitable containers. No heating steps are required by this process. If, however, a component is used which requires melting, the premelting of this component will be required.

20

Example 3: General Method #3 of Making Compositions

The solvent components such as the volatile silicone (for example, cyclomethicone), emollients (such as non-volatile silicone (for example, phenyltrimethicone), C12-15 alkyl benzoate, neopentyl glycol diheptanoate and isopropyl myristate) are added to a large capacity mixer equipped with a mechanical

25 stirrer and blended for about 5 minutes or until a homogeneous dispersion is formed. The antiperspirant active is then added as a dry powder with continuous mixing. This mixture is mixed for about 20 minutes or until a homogeneous dispersion is formed. The elastomer is then added and mixing is continued for about 10 minutes or until a homogeneous, white creamy paste is formed. At this point, the polyethylene beads are

added and mixing is continued for another 20 minutes. If fragrance is to be added, it is done at this point and mixed until well blended or for about 5 minutes. The resulting soft solid is then pumped through a homogenizer (KINEMATIC – MT 61 Megatron unit homogenizer). Multiple passes through the homogenizer are used (usually about 1-4). It has been shown through experimentation that 4 passes is optimal for the stability of the final product with regard to separation/syneresis. At 1-4 passes the product exhibits a syneresis of less than 8%, preferably less than 5% as evaluated by the process described in Example 5, below. The final product may be placed in suitable containers. No heating steps are required by this process. If, however, a component is used which requires melting, the premelting of this component will be required.

Example 4: General Method #4 of Making Compositions

When the elastomer content is in the low range of the composition and the fluids are of a percentage in the range of 18-23% by weight based on the total weight of the composition, the following process can be used. The cyclomethicone, dimethicone, neopentyl glycol diheptanoate, diethyl phthalate and isopropyl myristate are weighed separately and added sequentially to a properly sized stainless steel container. The ingredients are blended for about 5 minutes or until a homogeneous mixture is formed. The antiperspirant active is then slowly added while mixing is continued. Mixing can be done by an axial flow impeller with adequate power. Mixing is then continued for an additional 10 minutes. Next the polyethylene powder is added slowly with continued mixing. After the addition is completed, mixing is continued for an additional 10 minutes. This mixture is called the active phase. It is then added to a scraped wall mixing vessel with counter rotating blades (Lee, Agi or similar type) into which the formula amount of elastomer has already been charged. This is mixed for 20 minutes. If fragrance is used it is added at this point and mixing is continued for an additional 5 minutes. The soft solid product is then homogenized as described in Example 3. The product may then be transferred to suitable containers. No heating steps are required by this process. If, however, a component is used which requires melting, the premelting of this component will be required.

Example 5: Stability Evaluation

Samples (10.0g) of formulated product are placed in a 25 cm³ vial. The sample is incubated at a temperature of 50 degrees C. for 3 days and then centrifuged for 20 minutes at speed of 3900 rpm on a Centra CL2 centrifuge (from International Equipment Co., Needham Heights, Mass.). Any liquid remaining on top of the product sample was removed with a pipette and weighed. Percent syneresis is calculated as:

(weight of liquid removed after centrifugation) X 100/10.0

Normally it was desired to obtain syneresis levels of less than 8% and preferably less than 5% as described above. By way of comparison, the percent syneresis of a commercially available soft solid (Secret® Platinum powder fresh scent soft solid, a wax bases product containing 19% aluminum zirconium trichlorohydrate gly (anhydrous), cyclopentasiloxane, dimethicone, tribehenin, C18-36 acid triglyceride and fragrance) exhibited a percent syneresis of about 19.79%.

Examples 12, 17, 18, 19, 20, 21, 22, 23 and 24

Examples 12, 17, 18, 19, 20, 21, 22, 23 and 24 were made using the process described in Example 2 with the amounts of ingredients listed in TABLE D and TABLE E. Syneresis was measured using the process described in Example 3.

TABLE D

Ingredient	Ex. 12	Ex. 17	Ex. 18	Ex. 19
Elastomer (KSG-15)	262.50g (52.50%)	262.50g (52.50%)	262.50g (52.50%)	262.50g (52.50%)
Antiperspirant active (AZZ-902 SUF)	125.00g (25.00%)	125.00g (25.00%)	125.00g (25.00%)	115.00g (23.00%)
Phenyl trimethicone (Dow Corning 556)	5.00g (1.00%)	10.00g (2.00%)	5.00g (1.00%)	5.00g (1.00%)
Neopentyl glycol diheptanoate	25.00g (5.00%)	25.00g (5.00%)	25.00g (5.00%)	25.00g (5.00%)
Cyclomethicone (D5) DC-245	32.50g (6.50%)	27.50g (5.50%)	37.50g (7.50%)	47.50g (9.50%)
Isopropyl myristate	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)
Dimethicone (DC - 200, 1.5 centistokes)	10.00g (2.00%)	10.00g (2.00%)	10.00g (2.00%)	10.00g (2.00%)
Polyethylene (MICROTHENE FN 510)	30.0g (6.00%)	30.00g (6.00%)	25.00g (5.00%)	25.00g (5.00%)
Fragrance	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)
Total	500g (100%)	500.00g (100%)	500.00g (100%)	500.00g (100%)
% Syneresis	1.07	0.30	1.27	2.37

5

TABLE E

Ingredient	Ex. K20	Ex. K21	Ex. K22	Ex. K23	Ex. K24
Elastomer (KSG-15)	262.50g (52.50%)	262.50g (52.50%)	262.50g (52.50%)	262.50g (52.50%)	262.50g (52.50%)
Antiperspirant active (AZZ-902 SUF)	125.00g (25.00%)	125.00g (25.00%)	125.00g (25.00%)	125.00g (25.00%)	125.00g (25.00%)
Phenyl trimethicone (Dow Corning 556)	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)
Neopentyl glycol diheptanoate	25.00g (5.00%)	25.00g (5.00%)	25.00g (5.00%)	25.00g (5.00%)	25.00g (5.00%)
C12-15 alkyl benzoate (FINSOLV TN)	10.00g (2.00%)	0	10.00g (2.00%)	20.00g (4.00%)	30.00g (6.00%)
Cyclomethicone (D5) DC-245	22.50g (4.50%)	32.50g (6.50%)	22.50g (4.50%)	12.50g (2.50%)	2.50g (0.50)
Isopropyl myristate	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)
Dimethicone (DC 200, 1.5 centistokes)	10.00g (2.00%)	10.00g (2.00%)	10.00g (2.00%)	10.00g (2.00%)	10.00g (2.00%)
Polyethylene (MICROTHENE FN 510)	30.00g (6.00%)			30.00g (6.00%)	30.00g (6.00%)
Polyethylene (Performalene™)		30.00g (6.00%)	30.00g (6.00%)		
Fragrance	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)	5.00g (1.00%)
Total	500g (100%)	500.00g (100%)	500.00g (100%)	500.00g (100%)	500.00g (100%)
% Syneresis	0.44	0.00	0.00	0.00	2.37

Claims

We Claim:

1. A low residue antiperspirant and/or deodorant compositions comprising:
 - 5 (a) 40-75% of a volatile silicone;
 - (b) 0.1-20% of an emollient which may be a single emollient or a mixture of emollients;
 - (c) 0.5-6% (on a solids basis) of a dimethicone/vinyldimethicone crosspolymer composition made by reacting a polymethylhydrogensiloxane with an alpha,
10 omega-divinylpolydimethyl siloxane;
 - (d) 0.1-20% of an antiperspirant active based on an anhydrous, buffer-free basis;
 - (e) 2-15% of polyethylene beads having a particle size in the range of 5-40 microns and a density in the range of 0.91-0.98 g/cm³;
 - (f) 0-5% antimicrobial agent; and
 - 15 (g) 0-5% fragrance;wherein all percents are in percents by weight based on the total weight of the composition unless otherwise indicated.
2. An antiperspirant and/or deodorant composition according to Claim 1
20 comprising 45-60 weight % of a volatile silicone.
3. An antiperspirant and/or deodorant composition according to Claim 1 or 2 wherein the volatile silicone is cyclomethicone.
- 25 4. An antiperspirant and/or deodorant composition according to Claim 1 comprising 2-18 weight % of an emollient wherein the emollient comprises one or more members selected from the group consisting of:

(a) fats and oils which are the glyceryl esters of fatty acids or triglycerides of

Formula III:



Formula III.

10 wherein each of R^1 , R^2 , and R^3 may be the same or different and have a carbon chain length (saturated or unsaturated) of 7 to 30;

(b) hydrocarbons;

(c) esters of formula $R^4\text{CO-OR}^5$, wherein the chain length for each of R^4 and R^5 is from 7 to 30 and can be saturated or unsaturated, straight chained or

15 branched;

(d) saturated and unsaturated fatty acids having a formula $R^6\text{COOH}$ wherein R^6 has a carbon chain between 7-30 and can be straight or branched chain;

(e) saturated and unsaturated fatty alcohols having a formula $R^7\text{COH}$ wherein R^7 has a carbon chain between 7-30 and can be straight or branched chain;

20 (f) lanolin and its derivatives;

(g) alkoxyated alcohols wherein the alcohol portion is selected from aliphatic alcohols having 2-18 and more particularly 4-18 carbons, and the alkylene portion is selected from the group consisting of ethylene oxide, and propylene oxide having a number of alkylene oxide units from 2-53;

25 (h) silicones and silanes which are members of the group consisting of polymers of silicon/oxygen having general structures:

(1) $(R^{10})_3\text{SiO}(\text{Si}(R^{11})_2\text{O})_x\text{Si}(R^{12})_3$ where R^{10} , R^{11} and R^{12} can be the same or different and are each independently selected from the group consisting of phenyl and C1-C60 alkyl;

(2) $\text{HO}(\text{R}^{14})_2\text{SiO}(\text{Si}(\text{R}^{15})_2\text{O})_x\text{Si}(\text{R}^{16})_2\text{OH}$, where R^{14} , R^{15} and R^{16} can be the same or different and are each independently selected from the group consisting of phenyl and C1-C60 alkyl; or

- 5 (3) organo substituted silicon compounds of formula $\text{R}^{17}\text{Si}(\text{R}^{18})\text{OSiR}^{19}$ which are not polymeric where R^{17} , R^{18} and R^{19} can be the same or different and are each independently selected from the group consisting of phenyl and C1-C60 alkyl optionally with one or both of the terminal R groups also containing an hydroxyl group; and
- (i) mixtures and blends of two or more of (a)-(h).

10

5. An antiperspirant and/or deodorant composition according to Claim 4 wherein the emollient is one or more members selected from the group consisting of dimethicone, dimethiconol behenate, C_{30-45} alkyl methicone, stearoxytrimethylsilane, phenyl trimethicone and stearyl dimethicone.

15

6. An antiperspirant and/or deodorant composition according to Claim 1 comprising 1-6 weight % of the dimethicone/vinyldimethicone crosspolymer composition.

20

7. An antiperspirant and/or deodorant composition according to Claim 1 comprising 2-5 weight % of the dimethicone/vinyldimethicone crosspolymer composition.

8. An antiperspirant and/or deodorant composition according to Claim 1

25 comprising 10-20 weight % of an antiperspirant active.

9. An antiperspirant and/or deodorant composition according to Claim 1 comprising 2-10 weight % of polyethylene beads.
- 5 10. An antiperspirant/deodorant composition according to Claim 1 which is made as a soft solid and which comprises:
- (a) 1-20 weight % of cyclomethicone;
 - (b) 1-20 weight % of an emollient component comprising 1-8% of C12-15 alkyl benzoate; 0.5-5% neopentyl glycol diheptanoate; 0.5-2% isopropyl myristate; 0.4-10 1.5% of phenyltrimethicone;
 - (c) 40-60 weight % of a 4-10% dimethicone/vinyldimethicone crosspolymer composition in cyclomethicone;
 - (d) 15-25 weight % antiperspirant active;
 - (e) 2-10 weight % polyethylene beads; and
 - 15 (f) optionally 0.5-1.5 weight % fragrance.

THIS PAGE BLANK (USPTO)

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
11 October 2001 (11.10.2001)

PCT

(10) International Publication Number
WO 01/74306 A3

- (51) International Patent Classification⁷: A61K 7/48, 7/32 (74) Agent: MIANO, Rosemary, M.: Colgate-Palmolive Company, 909 River Road, P.O. Box 1343, Piscataway, NJ 08855-1343 (US).
- (21) International Application Number: PCT/US01/10330
- (22) International Filing Date: 29 March 2001 (29.03.2001)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data:
60/194,373 4 April 2000 (04.04.2000) US
09/712,378 14 November 2000 (14.11.2000) US
- (71) Applicant: COLGATE-PALMOLIVE COMPANY
[US/US]: 300 Park Avenue, New York, NY 10022 (US).
- (72) Inventors: MATTAL, Jairajh: 280 River Road, Apt. 21B, Piscataway, NJ 08854 (US). GUENIN, Eric, P.: 14 Arvida Drive, Pennington, NJ 08534 (US). AFFLITTO, John: 2 Jay Drive, Brookside, NJ 07926 (US). HOGAN, John: 242B Pleasantview Drive, Piscataway, NJ 08854 (US). JONAS, John: 96 West End Avenue, Summit, NJ 07901-1222 (US). LEE, Wilson: 51 Hearthstone Road, Bloomfield, NJ 07003 (US). LINN, Elizabeth: 621 Fifth Avenue, Lyndhurst, NJ 07071 (US). MUNSAYAC, Rosemary: 5 Tenney Road, West Orange, NJ 07052 (US). TANG, Xiaozhong: 190 Crestview Road, Bridgewater, NJ 08807 (US).
- (81) Designated States (*national*): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.
- (84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).
- Published:
— with international search report
- (88) Date of publication of the international search report:
16 May 2002
- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

(54) Title: STABLE AND EFFICACIOUS SOFT SOLID PRODUCT

(57) Abstract: A low residue, stable antiperspirant and/or deodorant composition is disclosed which comprises: (a) a dimethicone/vinyldimethicone crosspolymer composition made by reacting a polymethylhydrogensiloxane with an alpha, omega-divinylpolydimethyl siloxane for which the dimethicone/vinyldimethicone crosspolymer composition is used at a concentration of 0.5-10 % in cyclomethicone; (b) polyethylene beads having a density in the range of 0.91-0.98 grams/cm³ and a particle size in the range of 5-40 microns, wherein the polyethylene beads are used in an amount of at least 2 % by weight based on the total weight of the composition; (c) a volatile silicone; (d) an emollient or mixture of two or more emollients; and (e) an effective amount of an antiperspirant active material.

WO 01/74306 A3

INTERNATIONAL SEARCH REPORT

Int. Application No
PCT/US 01/10330

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A61K7/48 A61K7/32

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 A61K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

EPO-Internal, PAJ, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
E	WO 01 74325 A (COLGATE PALMOLIVE CO) 11 October 2001 (2001-10-11) claims	1-10
E	WO 01 51020 A (UNILEVER PLC ; LEVER HINDUSTAN LTD (IN); UNILEVER NV (NL)) 19 July 2001 (2001-07-19) page 56; examples 64-66	1-10
X	US 5 547 302 A (DORNBUSCH ARTHUR H ET AL) 20 August 1996 (1996-08-20) column 9; example	1-10
P, X	US 6 136 302 A (JUNEJA PREM SAGAR ET AL) 24 October 2000 (2000-10-24) column 9; table 2	1-10

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

* Special categories of cited documents:

- *A* document defining the general state of the art which is not considered to be of particular relevance
- *E* earlier document but published on or after the international filing date
- *L* document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
- *O* document referring to an oral disclosure, use, exhibition or other means
- *P* document published prior to the international filing date but later than the priority date claimed

T later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

X document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

Y document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

G document member of the same patent family

Date of the actual completion of the international search

21 January 2002

Date of mailing of the international search report

29/01/2002

Name and mailing address of the ISA
European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax (+31-70) 340-3016

Authorized officer

Couckuyt, P

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 5 654 362 A (SCHULZ JR WILLIAM JAMES ET AL) 5 August 1997 (1997-08-05) cited in the application the whole document -----	1-10
Y	US 5 069 897 A (ORR THOMAS V) 3 December 1991 (1991-12-03) examples -----	1-10
A	EP 0 485 012 A (PROCTER & GAMBLE) 13 May 1992 (1992-05-13) the whole document -----	1
A	US 5 989 531 A (MOGHE BHALCHANDRA ET AL) 23 November 1999 (1999-11-23) the whole document -----	1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 01/10330

Patent document cited in search report		Publication date	Patent family member(s)	Publication date
WO 0174325	A	11-10-2001	WO 0174325 A2	11-10-2001
WO 0151020	A	19-07-2001	AU 3369101 A WO 0151020 A1 US 2001018045 A1	24-07-2001 19-07-2001 30-08-2001
US 5547302	A	20-08-1996	AU 2954395 A EP 0772410 A1 JP 10503682 T WO 9603899 A1	04-03-1996 14-05-1997 07-04-1998 15-02-1996
US 6136302	A	24-10-2000	AU 1835000 A WO 0048564 A1	04-09-2000 24-08-2000
US 5654362	A	05-08-1997	NONE	
US 5069897	A	03-12-1991	NONE	
EP 0485012	A	13-05-1992	AT 133854 T CA 2054825 A1 DE 69116982 D1 DE 69116982 T2 EP 0485012 A1 US 5298236 A	15-02-1996 09-05-1992 21-03-1996 12-09-1996 13-05-1992 29-03-1994
US 5989531	A	23-11-1999	AU 1518300 A BR 9915298 A CZ 20011658 A3 EP 1128803 A1 WO 0028956 A1	05-06-2000 07-08-2001 12-09-2001 05-09-2001 25-05-2000